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EPA Region 10
Deemed ReleasableDATA VALIDATION REPORT
YAKIMA AGRICULTURAL RESEARCH LABORATORY

The following report presents a summary of the data validation review of analytical results for 45 soil samples, 2 water samples, and 2 wipe samples from the Yakima Agricultural Research Laboratory in Yakima, Washington. Samples were collected during 1990, 1991, and 1992. Samples were analyzed by Biospherics, Inc., of Beltsville, Maryland, and North Creek Analytical of Bothell, Washington. Sample numbers, collection dates (if available), and analyses conducted on each sample are shown in tables 1, 2, and 3.

DATA QUALIFICATIONS

Analytical results were reviewed following procedures presented in *Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses* (USEPA, 1988a) and *Laboratory Data Validation Functional Guidelines for Evaluating Pesticides and PCBs Analyses* (USEPA, 1988b).

Timeliness

All samples listed in table 1 were extracted (if appropriate) and analyzed within EPA holding time criteria, except for those noted below. The VOC analysis performed on 8/19/92 for sample DP-5 was the initial analysis performed outside of holding time. The VOC analyses performed on 8/19/92 and 8/26/92 for samples DP-4 and DP-5, respectively, were the reanalyses.

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Sample Number	Date Collected	Analysis	Date Extracted	Date Analyzed	Holding Time	Holding Time Criteria
DP-4	7/27/92	VOCs	--	8/19/92	23 days	14 days
DP-5	7/27/92	VOCs	--	8/19/92	23 days	14 days
DP-5	7/27/92	VOCs	--	8/26/92	30 days	14 days

Table 1
Sample Summary - July 1992
Yakima Agricultural Research Laboratory

Sample Number	Date Collected	Matrix	Analyses ¹
DP-1	7/27/92	Soil	8080, 8240, 8150, 8140
DP-2	7/27/92	Soil	8080, 8240, 8150, 8140
DP-3	7/27/92	Soil	8080, 8240, 8150, 8140
DP-4	7/27/92	Water	8080, 8240
DP-5	7/27/92	Water	8080, 8240
DF-6	7/27/92	Soil	8080, 8240, 8150, 8140
DF-7	7/27/92	Soil	8080, 8240, 8150, 8140
DF-8	7/27/92	Soil	8080, 8240, 8150, 8140
DF-9	7/27/92	Soil	8080, 8240, 8150, 8140
DF-10	7/27/92	Soil	8080, 8240, 8150, 8140
DF-11	7/27/92	Soil	8080, 8240, 8150, 8140
DF-12	7/27/92	Soil	8080, 8240, 8150, 8140
DF-13	7/27/92	Soil	8080, 8240, 8150, 8140
DP-6MS	7/27/92	Soil	8080, 8240, 8150, 8140
DF-6MDS	7/27/92	Soil	8080, 8240, 8150, 8140

¹ EPA methods 8080 = Pesticides and PCBs, 8240 = volatile organic compounds (VOCs), 8150 = chlorinated herbicides, 8140 = organophosphorus pesticides. These analyses were performed by North Creek Analytical, Inc. Bothell, Washington.

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Table 2
Sample Summary - June 1991
Yakima Agricultural Research Laboratory

Sample Number	Date Analyzed ¹	Matrix	Analyses
91061008-1	6/26/91	Soil	8080 ²
91061008-2	6/26/91	Soil	8080 ²
91061205-1	6/26/91	Soil	8080 ²
91061205-2	6/26/91	Soil	8080 ²
91061205-3	6/26/91	Soil	8080 ²
91061302-5	6/26/91	Soil	8080 ²
91061302-6	6/26/91	Soil	8080 ²
91061321-1	6/29/91	Soil	8080 ²
91061321-2	6/29/91	Soil	8080 ²
91061204-1	6/29/91	Soil	8080 ²
91061204-2	6/29/91	Soil	8080 ²
91061302-1	6/29/91	Soil	8080 ²
91061302-2	6/29/91	Soil	8080 ²
91061302-3	6/29/91	Soil	8080 ²
91061302-4	6/29/91	Soil	8080 ²
61008-1MS	6/25/91	Soil	8080 ²
61008-1MSD	6/26/91	Soil	8080 ²
61302-4MS	6/26/91	Soil	8080 ²
61302-4MSD	6/26/91	Soil	8080 ²

1. Sample collection date is not known.
2. EPA methods 8080 = Pesticides and PCBs. These analyses were performed by Biospherics, Inc., Beltsville, Maryland.

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Table 3
Sample Summary - October 1990
Yakima Agricultural Research Laboratory

Sample Number	Date Analyzed ¹	Matrix	Analyses
9019695,20,1	10/29/90	Soil	8080 (PCBs only) ²
9019696,20,1	10/29/90	Soil	8080 (PCBs only) ²
9019697,20,1	10/29/90	Soil	8080 (PCBs only) ²
9019698,20,1	10/29/90	Soil	8080 (PCBs only) ²
9019707,20,1	10/29/90	Soil	8080 (PCBs only) ²
9019560,40,2.04	10/29/90	Soil	8080 (PCBs only) ²
9019699,20,1	10/29/90	Soil	8080 (PCBs only) ²
9019700,20,1	10/29/90	Soil	8080 (PCBs only) ²
9019701,20,1	10/29/90	Soil	8080 (PCBs only) ²
9019702,20,1	10/29/90	Soil	8080 (PCBs only) ²
90102903-3	10/29/90	Soil	8080 (PCBs only) ²
90102903-1	10/30/90	Soil	8080 (PCBs only) ²
9019339	10/30/90	Soil	8080 (PCBs only) ²
9019340	10/30/90	Soil	8080 (PCBs only) ²
9019341	10/30/90	Soil	8080 (PCBs only) ²
9019548	10/30/90	Soil	8080 (PCBs only) ²
9019549	10/31/90	Soil	8080 (PCBs only) ²
9019554	10/31/90	Wipe	8080 (PCBs only) ²
9019555	10/31/90	Wipe	8080 (PCBs only) ²

¹ Sample collection date is not known.
² EPA methods 8080 = PCBs only. These analyses were performed by Biospherics, Inc., Beltsville, Maryland.

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An estimated data qualifier (J) was assigned to the results for VOCs and an estimated data qualifier (UJ) was assigned to the detection limits in samples DP-4 and DP-5 based on exceedance of holding time criteria.

Holding times could not be assessed for the samples listed in tables 2 and 3 because sample collection dates were not available.

GC/MS Tuning

Raw data and summary forms for EPA method 8240 for gas chromatography/mass spectrometry (GC/MS) instrument tuning were evaluated for compliance with USEPA QC requirements. All ion abundance criteria for bromofluorobenzene (BFB) were within USEPA QC criteria (USEPA, 1991).

Calibration

Raw data and summary forms for initial and continuing calibration of VOCs, pesticides and PCBs, chlorinated herbicides, and organophosphorus pesticides analyses were reviewed to evaluate frequency, average response factors (RFs) for initial and continuing calibration, percent relative standard deviation (%RSD) between initial calibration RFs, and percent difference (%D) between initial calibration average RF and continuing calibration RF.

Initial calibration QC criteria for VOCs, pesticides and PCBs, chlorinated herbicides, and organophosphorus pesticides were within USEPA QC criteria (USEPA, 1988a and 1988b), with the following exceptions.

The EPA method 8240 initial calibration average RFs for dichlorodifluoromethane (0.003; calibration on August 7, 1992) and acetone (0.004; calibration on August 19, 1992) were less than the minimum QC criteria of 0.05. All detected results for acetone (EPA method 8240) for the July 1992 data were assigned an estimated data qualifier (J), and all non-detected results for dichlorodifluoromethane were assigned an unusable data qualifier (R) in accordance with USEPA data validation procedures (USEPA, 1988a). The initial calibration %RSD for the 1992 data for acetone also exceeded the maximum QC criteria of 30% RSD.

The initial calibration %RSD for DDT (EPA method 8080) for the July 1992 data exceeded the 10% QC criteria. All detected results for DDT were assigned an estimated data qualifier (J).

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The initial calibration %RSD for endrin, DDT, b-BHC, d-BHC, heptachlor epoxide, endosulfan I, DDE, dieldrin, endosulfan II, DDD, endrin aldehyde, endosulfan sulfate, endrin ketone, methoxychlor (MOC), dibutylchloroendate (DBC), and tetrachloro-meta-xylene (TCM) for the June 1991 data exceeded the 10% QC criteria. All detected results for these compounds were assigned an estimated data qualifier (J).

Continuing calibration QC criteria for VOCs, pesticides and PCBs, chlorinated herbicides, and organophosphorus pesticides were within USEPA QC criteria (USEPA, 1988a and 1988b), with the following exceptions.

The EPA method 8240 continuing calibration RFs for acetone and dibromomethane (0.04 and 0.003; calibration on August 19, 1992) were less than the minimum QC criteria of 0.05. All detected results for acetone (analyzed on August 19, 1992) were assigned an estimated data qualifier (J), and all nondetected results for dibromomethane (analyzed on August 19, 1992) were assigned an unusable data qualifier (R). The %D for acetone (for August 10, 1992) exceeded the maximum QC criteria of 25%. Acetone results for August 10, 1992 were assigned an estimated data qualifier (J).

The EPA method 8080 continuing calibration standard analyzed on August 20, 1992 did not contain DDE and DDT, which were detected in the samples.

The EPA method 8080 continuing calibration %D for June 26, 1991 exceeded the 15% QC criteria for all pesticides except a-BHC, b-BHC, lindane, aldrin, dieldrin, and endrin aldehyde. For June 28 and 29, 1991, the %D exceeded the 15% QC criteria for all pesticides except a-BHC, b-BHC, lindane, and endrin ketone. For June 29, 1991, the % difference exceeded the 15% QC criteria for all pesticides, except a-BHC. All detected results for these compounds were assigned an estimated data qualifier (J).

Method Blank Results

Laboratory method blank analyses were conducted at the required frequency for all analytical methods. No compounds were detected in method blanks at concentrations greater than quantitation limits, with the following exceptions.

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Blank Identification	Analysis	Compound	Blank Concentration (µg/kg)	Associated Samples	Sample Concentration (µg/kg)
BLK081092	VOCs	Acetone	15	DP-1	7.0U
				DP-2	4.2U
				DP-3	2.5U
				DF-6	2.0U
				DF-7	1.5U
				DF-8	1.4U
				DF-9	1.3U
				DF-11	1.2U
				DF-12	1.0U
				DF-13	0.96U
		2-butanone	0.56	DP-2	0.58U
				DP-3	0.55U
				DF-6	0.52U
				DF-7	0.54U
				DF-10	0.68U
				DF-12	0.54U
				DF-13	0.56U

The concentrations of acetone and 2-butanone in the associated samples listed above were less than ten times the acetone and 2-butanone concentrations in method blank BLK081092. In accordance with USEPA data validation procedures (USEPA, 1988a), an undetected data qualifier (U) was assigned to the results for acetone and 2-butanone in the samples listed above.

Sample preparation logs showing amounts of sample extracted were not available for the EPA method 8080 analyses performed in 1990 and 1991. Therefore, conclusive comparison of method blank analyte levels to sample analyte levels were not made.

Matrix Spike/Matrix Spike Duplicate Results

All matrix spike/matrix spike duplicate (MS/MSD) results were within QC criteria for percent recovery (%R) and percent relative percent difference (%RPD) between MS and MSD results with the following exceptions:

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					QC Limits	
Sample Number	Compound	MS %R	MSD %r	%RPD	%R	%RPD
DF-13	1,1-Dichloroethene (1,1-DCE)	38	48	22	43-98	19
DF-6	DDT	224	241	2	38-127	27
DF-6	Heptachlor	90	102	13	84-115	10
DF-6	Endrin	106	124	21	56-121	21
61008-1	Endrin	78	120	42	(not provided)	(not provided)
61302-4	Dieldrin	500	400	22	(not provided)	(not provided)
61302-4	DDT	160	150	6	(not provided)	(not provided)

No data qualifiers were assigned to sample results based on MS/MSD analyses. However, MS/MSD recovery results for DDT for soils collected in 1991 and 1992, and for dieldrin for soils collected in 1991, indicate that sample results maybe biased high.

Surrogate Recoveries

Raw data and summary forms for surrogate compounds were evaluated for compliance with USEPA and method QC criteria.

For samples listed in table 2, the laboratory did not report the amounts of the surrogate added; therefore, surrogate recoveries were not calculated. The samples listed in table 3 did not contain an identified surrogate compound.

The percent recoveries for surrogate compounds in the remaining samples (table 1) were within QC criteria for all analyses with the following exceptions:

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Sample Number	Analysis	Surrogate Compound	%R
DP-4	Pesticides/PCBs	tetrachloro-m-xylene	25
DP-5	Pesticides/PCBs	tetrachloro-m-xylene	15
BLK080392W	Pesticides/PCBs	tetrachloro-m-xylene	34
DP-1	Chlorinated Herbicides	2,4-Dichlorophenyl Acetic Acid	27

In accordance with USEPA data validation guidelines (USEPA, 1988b), an estimated data qualifier (J) was assigned to the affected sample results greater than the quantitation limits and an estimated data qualifier (UJ) was assigned to sample quantitation limits in all pesticides and PCBs in samples DP-4 and DP-5 based on low surrogate recoveries for these samples and the method blank. The laboratory did not reanalyze the samples because there was not enough sample volume to reextract.

Internal Standards Performance

Raw data and summary forms for internal standards (IS) retention times and area counts for VOC analyses were evaluated for compliance with USEPA QC criteria (USEPA, 1991). All IS retention times and area counts were within QC limits.

Identification, Quantitation, Verification, and Reporting Limits

Mass spectra, quantitation reports, and quantitation limits for VOC analyses performed in 1992 were reviewed for compliance with QC criteria (USEPA, 1988a, 1991). No errors were found with compound identification, quantitation, or reporting.

Retention times for detected pesticides were compared with retention time windows for the July 1992 data. Detected compounds were outside the retention time window for the primary column in several samples. However, the detected compounds outside the retention time window for the primary column were within the retention time windows for either the confirmation column, the diluted reanalyzed sample, or both except for DDT in samples DP-4, DP-5, and DF-6, and DDE in sample DF-6. Qualifiers were not

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assigned because these compounds were marginally outside the retention time windows.

Retention time windows were not provided by the laboratory for pesticides and PCB analyses for the 1990 and 1991 data.

OVERALL ASSESSMENT OF DATA

Data precision was evaluated by a comparison of MS/MSD results with method QC criteria. The precision of the VOCs, pesticides and PCBs, chlorinated herbicide, and organophosphorus pesticides data for July 1992 were judged to be acceptable for the intended use in site characterization. Accuracy of these sample data was evaluated using MS/MSD and blank sample results. The accuracy of the July 1992 analyses was judged to meet the method QC criteria.

The following information required for data validation according to the USEPA Functional Guidelines (1988a and 1988b) were not available for the pesticides and PCBs analyses performed in 1990 and 1992 (tables 2 and 3): chains-of-custodies, retention time window summaries, sample preparation logs, and identification of primary and secondary columns. Therefore, the data could not be validated according to the EPA Functional Guidelines and no judgement is made regarding the acceptability of the data for its intended use.

The data obtained in 1992 (table 1) are judged to be acceptable for their intended use. The usefulness of the results may be modified by assignment of the following data qualifiers to individual compounds and sample results:

J—The associated numerical value is an estimated quantity.

U—The material was analyzed for, but was not detected at a concentration greater than the associated value. The associated numerical value is the sample quantitation limit or sample detection limit.

UJ—The material was analyzed for, but was not detected. The associated numerical value is the estimated quantitation limit.

R—The data are unusable (analyte may or may not be present). Resampling and reanalysis are necessary for verification.

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N—Presumptive evidence of the presence of material.

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- U.S. Environmental Protection Agency. 1986. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods. USEPA, Office of Solid Waste and Emergency Response. EPA 530/SW-846.
- U.S. Environmental Protection Agency. 1988a. Laboratory Data Validation Functional Guidelines for Evaluating Organics Analyses. Prepared for the Hazardous Site Evaluating Division, U.S. Environmental Protection Agency. Prepared by the USEPA Data Review Work Group.
- U.S. Environmental Protection Agency. 1988b. Laboratory Data Validation Functional Guidelines for Evaluating Pesticides and PCBs Analyses. Prepared for the Hazardous Site Evaluation Division, U.S. Environmental Protection Agency. Prepared by the USEPA Data Review Work Group.
- U.S. Environmental Protection Agency. 1991. U.S. Environmental Protection Agency Contract Laboratory Program, Statement of Work for Inorganics and Organics Analyses, Multi-media, Multi-concentration. USEPA, Washington, D.C.

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